CHEMICAL PROCESSES

Focus: This lab consists of three experiments: one related to energy loss which can affect environmental systems, or chemical processing operations; and two related to chemical separation processes.

Part 1: Energy Transfer/Loss

Introduction

Effective transfer and conversion of energy is an extremely important consideration in almost all products of engineering—engines, drive trains, heating, air conditioning, chemical manufacturing, materials production, electricity production—the list is endless. Efficiency translates into $$$. How the energy is supplied, from what source, how it is moved to another place, and what losses are incurred in the process are all contributors to efficiency.

In this experiment, we're interested in energy as heat. Energy as heat is transferred by three mechanisms: conduction, convection, and radiation. Conduction occurs when two static systems at different temperatures are brought in contact with one another. Heat flows from the higher temperature system to the lower temperature system through the materials themselves. For example, your hands holding a cup of hot soup become warm by being in contact with the hot cup. Convection is the physical transport of heat via fluid medium. It can be "natural convection" associated with density differences—the plume of warm air over a hotplate--or it can be forced convection as in a "convection" oven, which uses a fan to circulate air. Finally, radiation is the transport of energy as electromagnetic waves (with no need for a transporting medium at all). These waves are absorbed by the receiving body and are, hence, warmed. The warmth of a roaring fire is an example.

Description of Experiment

In this experiment you will heat a pot of water with a hotplate (Calrod) and deduce the efficiency of transferring the input energy (electrical) to the output product (hot water). Electrical energy flows through a resistance element atop the Calrod to cause "Ohmic heating" or "resistance heating". This element, through conduction, convection, and radiation heats its surroundings—the pot of water, the Calrod unit itself, and the air. The idea is to convert as much of this electrical energy to heat that directly transfers to the water. You are interested in the efficiency of this energy transfer between the electrical energy input to the Calrod unit and the temperature rise of the water. And, you are interested in the effect of putting a lid on a pot of heating water.

Procedure

1. Plug the "Kill A Watt"monitor into an electrical outlet. Press the appropriate button so that "watt" shows on the screen.
2. Pour into the pot 500 cc of water, weigh the pan and water, and place it on the hotplate.
3. Record the room temperature and the temperature of the water.
4. Set the heating dial on the hotplate on high and connect it to the outlet terminal of the "Kill A Watt".
5. Record the watts and time every 30 seconds until the water temperature reaches 80 to 90C (the watt readings will be relatively constant).
6. Shut off the electrical input.
7. Weigh the pot and water after the experiment is concluded.
8. Repeat the experiment with another heating unit, but this time put a lid on the pot. (Using a second, cold heating unit ensures that the initial conditions between the two experiments are similar.)

Questions and writeup

1. Calculate the total energy input (from the time you turned the heating unit on to the time you turned it off.) Watts * time = total energy. Make a plot of watts vs. time. (It'll probably be a relatively uninteresting plot.)
2. Calculate the energy increase in the water (1^0 C /cc = 1 cal.)
3. Calculate the efficiency as a function of time (energy increase in water/total input energy). (Consistent units would be nice.) If it's greater than 1.0, rethink your calculations. If it's less than 1.0, where did the remaining energy go? Try to think of all possible "losses". How much energy went into heating the pot? (specific heat of material x mass x heat rise).
4. Explain any differences you found between the before and after weights of pot and water
5. What difference did putting a lid on the pot make?
6. How might have the efficiencies been improved? Bigger heating element? Small pot? Try to envision all the heat transfer that is taking place.
7. Explain the efficiency vs.time plot.

Part 2: Paper Chromatography

Overview:

Chromatography separates different chemical substances—in this case colored dyes--by making use of their different flow rates while being transported by a diffusing solvent. Flow rates are defined as being the distance a solute (a chemical substance) travels divided by the distance traveled by the solvent. For example, one color in a solvent may move farther than another color in the same solvent even if they are given the same length of time to move.

If the end of a piece of absorbent paper with an ink pen line on it is placed in solvent, the solvent will be drawn up the surface of the paper by capillary action. As it passes the ink line, the ink will dissolve in the solvent and be carried along with the solvent (this is mass transport). The ink, though, will be differentially retarded by the paper. A competition for ink is set up between the solvent and the paper, and as a result, the ink will travel up the surface of the paper more slowly than the solvent. The relative strength of the ink's attraction for the paper and solvent varies between inks. If there is more than one ink in a given pen, each ink will travel at its own pace, and you will see each separate ink emerge. In addition, some solvents will dissolve certain inks better than others. When solvents are used that do not dissolve the ink well, most of the ink will be left behind at the original line and only a small amount will travel up the paper.

Procedure:

You will be given three pieces of filter paper (a coffee filter could have been used) that you should cut into rectangles about 4cm x 10 cm. Using the three pens provided, make a line segment about 0.75cm long with each of the pens about 1 cm from one end of the each paper strip. The distance between marks should be about 0.75cm; the end result should be a dashed line where each line segment has been made with a different pen. Make multiple passes so the ink is dark. Put several lines of the same color directly on top of each other rather than overlapping them. The objective is to provide sources of dye which can diffuse up the length of the strips. Let the ink dry while you make a horizontal pencil mark connecting the two ink lines and extending beyond them. Make another pencil mark near the other end of the strip. These will mark the end point of the experiment.

You will be conducting nine separate experiments: three with water as a solvent, three with methanol(POISON) as a solvent, and three with 2-propanol(POISON). Each of the ink marks is its own separate experiment. First pour solvent into the developing tank to a depth of about 1 cm. Attach the plain end of your prepared paper strip to a straightened paper clip. This wire will be used to suspend the filter paper over the solvent. Lower the paper into the solvent to a depth of 2-3mm, but not so deep that the ink marks are in the solvent. Cover the tanks with a piece of paper to minimize evaporation.

The solvent will rise, partially carrying the ink. Once the solvent reaches the pencil mark (which won't dissolve in the solvents), remove the paper strip from the tank and let it dry. Measure the farthest excursions of all ink lines on this "chromatogram". The ratio of the distance traveled by the ink to the distance traveled by the solvent is the flow rate for that ink in that solvent. Perform this experiment with water, methanol, and 2-propanol as solvents.

Write-up:

First, describe what happened, taking care to differentiate between different colors and different solvents. For the methanol and 2-propanol experiments, calculate flow rates from the purple ink and present a graph showing the flow rate of each component as a function of the proportion of 2-propanol in the solvent (approximately 100% for 2-propanol and 0% for methanol). Assuming that flow rate changes linearly with 2-propanol concentration, draw a line which represents the expected flow rate for each component in various concentrations. Deduce the expected equations of each of these lines.

Notice that for the purple pen, one ink component flows faster in 2-propanol while the other flows faster in methanol. If we were attempting to separate these two components, and we were really unlucky by choosing the wrong concentration of methanol, we could obtain no separation. Estimate the methanol concentration in 2-propanol of this most undesirable solvent?

Part 3: Distillation

Overview:

The objective of this experiment is to apply the process of distillation to a mixture of ethanol and water in order to obtain a new mixture with an increased concentration of ethanol. In addition, the results of the experiment will be used to approximate the equilibrium relationship between ethanol and water.

Distillation has been described as the "work-horse" of chemical engineering because of its widespread use in industry. Distillation is a type of separation process that is useful in separating components of a liquid mixture that have different boiling points.

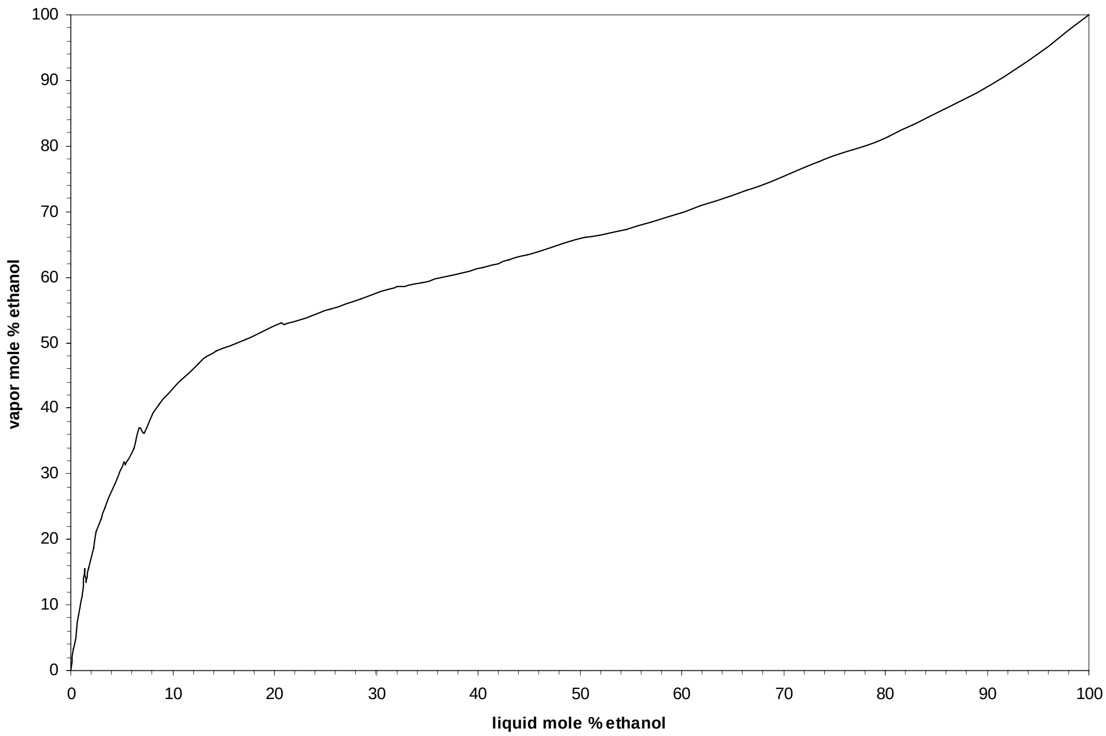
For example, consider a mixture consisting of two components, A and B, that have boiling points of 70°C and 100°C, respectively. When the mixture begins to boil, the vapor phase that is formed will be richer in A than the liquid phase. This is because A has a lower boiling point and vaporizes more easily than B. Therefore, A has a greater tendency to enter the vapor phase while B tends to remain in the liquid phase. Of course, if the entire mixture were allowed to boil away, the resulting vapor would have the same concentration of A and B as the original liquid mixture. However, if only a fraction of the liquid is allowed to boil, the vapor will contain a higher concentration of A than the original liquid mixture.

As the mixture continues to boil, the compositions of both the vapor and liquid phases change with time. Since A enters the vapor phase more quickly than B, the concentration of A in the liquid phase decreases while that of B increases. Also, as the temperature of the boiling liquid increases, more B vaporizes as time passes, and the concentration of B in the vapor phase also increases with time.

If some fraction of the original mixture vaporizes and the vapor is collected and allowed to cool and condense in a separate container, the new liquid mixture will have a higher concentration of A than the original did. With each repetition of this process, we would obtain a new liquid mixture with a higher A concentration than the previous mixture. This is basically how distillation works. It is just a series of vaporization and condensation processes that continues until a desired concentration is reached.

In general, an equilibrium stage in any type of unit operation process is defined as an area in which different phases are brought into close contact so that a component can be redistributed between the phases to equilibrium concentrations. In the simple batch distillation setup in this experiment, liquid in the 1000-mL flask is in close contact with the vapor phase that forms as the mixture boils, and this is the only place where this situation is present in the setup. Therefore, there is one equilibrium stage in this batch distillation. Assuming everything works ideally, the vapor phase is in equilibrium with the liquid in the flask at any given time.

The type of equilibrium information that is needed for analyzing this separation process is that which shows the vapor-liquid equilibrium relationship for an ethanol/water mixture. The most useful representation of the data is a y versus x graph, where y and x are the vapor and liquid phase concentrations, respectively, of the more volatile component, ethanol. Points on the curve depicted in such a graph represent the concentration of ethanol in the vapor and liquid phases at equilibrium. Along the equilibrium curve, pressure is constant, but temperature is different at each point. A picture of the ethanol/water y versus x graph is shown below.



<u>Procedure</u>:

This experiment involves small-scale batch distillation. The basic setup is shown to the right.

The original ethanol/water solution is placed in the 1000-mL flask. Heat to the mixture is provided by a hot plate. A rubber stopper caps the flask. The stopper contains two small holes: one for a thermometer, and one for a small piece of copper tubing to which latex tubing may be attached. Another rubber stopper with a small piece of copper tubing is plugged into the top of the Graham condenser. The flask and condenser stoppers are connected with latex tubing. The water jacket surrounding the glass coil must be connected to a water source (lower fitting) and sink (upper fitting) via latex tubing. The role of the condenser is to extract enough heat from the vapor produced in the flask to condense back into a liquid phase. (The condensed liquid is referred to as *distillate*.) The condensed liquid empties into collection

bottles at the bottom of the coil.

During the experiment, you will allow the flask mixture to boil. Vapors produced from boiling will escape the flask through the copper and latex tubing and will condense in the condenser to form the distillate. The distillate will drain into the collection beaker. Three 40-mL samples of distillate will be collected. In order to analyze this process quantitatively, it is necessary to know the concentration of ethanol in the distillate and in the flask mixture at the beginning and end of the experiment.

In order to determine the percent of ethanol in the various liquid volumes, a temperature / specific gravity / mass fraction correlation chart for the ethanol/water system in Perry's Chemical Engineers' Handbook is used. You will measure the specific gravity and temperature of a liquid and use these two pieces of data with the chart to determine the mass fraction of ethanol. There are five liquid samples for which you will need to determine the mass fraction of ethanol: the initial solution, three distillate samples, and the final mixture in the flask at the end of the experiment.

The specific gravity is measured using a hydrometer. To use the hydrometer, collect approximately 40 mL of liquid in a 50-mL graduated cylinder. Place the hydrometer in the liquid and give it a gentle push downward so that it bobs up and down. Once the hydrometer comes to a rest, read the specific gravity at the surface level of the liquid. When taking hydrometer readings, it is very important that the hydrometer is floating freely and not resting against the side of the graduated cylinder. Also, readings are very sensitive to temperature, so be sure to record the temperature of the liquid with each hydrometer reading.

Begin the experiment by preparing the initial 800-mL ethanol/water mixture. The mixture should be approximately 10% (by volume) ethanol. There are two important rules to follow while preparing the mixture:

1) Do not use the markings on the flask to measure volumes; they are not very exact.
2) Measure the ethanol and water **separately** using a graduated cylinder. Do not mix ethanol and water together in the graduated cylinder while measuring the volumes for the initial mixture; mixing will affect the volume readings.

To make the solution, measure the appropriate volumes of each liquid using a graduated cylinder. Pour measured volumes of liquid from the cylinder to the flask, and keep track of the total volume of the liquid using the cylinder's markings.

After making the solution, carefully swirl the flask to ensure that the ethanol and water are mixed well. (stir instead?) Now, measure the temperature and specific gravity of the initial solution. While doing this, be very careful not to lose any of your solution as this will affect your final calculations.

After determining the specific gravity and temperature of the initial solution, carefully pour the sample back into the flask, trying to be sure that none is lost. Place the rubber stopper (with the copper tubing in it) on the flask. The tubing should stick out past the stopper approximately ½" into the flask.

Start water flowing through the cooling jacket of the condenser. Make sure that the flow is from bottom to top. Otherwise, the coils may not be fully bathed in the cooling water. The flow should be a gentle, but steady stream.

Turn the hot plate setting to 9 and leave it at this setting for approximately 10 minutes. Then, turn the heat setting to 5 ½. The solution should not be boiling yet, but it will probably begin to boil within 8-10 minutes. Once the solution begins to boil, it should be about 3-5 minutes until you observe a distillate stream emptying into the collection bottle. Throughout the experiment, be sure that the flask mixture does not boil harshly.

After the bottle has collected approximately 40 mL of distillate, quickly replace it with an empty one. This needs to be done as rapidly as possible so that minimal distillate is lost. Cover and label the bottle containing the first distillate sample.

The temperature / specific gravity / mass fraction correlation chart only goes up to 40°C. Therefore, if the distillate is warmer than 40°C, you will need to allow it to cool before measuring its specific gravity. If it is already cool enough, it is a good idea to go ahead and measure its specific gravity while collecting the second distillate sample. To take the specific gravity measurement, pour the contents of the beaker into a 50-mL graduated cylinder. First, record the volume of the sample. This will be important for later calculations. (If you collected more then 50 mL of distillate, determine the volume using a larger graduated cylinder or measure the volume in sections with the 50-mL cylinder.) After recording the volume, use the 50-mL graduated cylinder and the hydrometer to determine the sample's specific gravity. (If the cylinder is too full, some of the liquid can be poured out after the volume is measured since it is not necessary to have the total volume present to measure specific gravity; specific gravity is not dependent on volume.)

Use the same procedure to collect the second distillate sample. The third distillate sample is collected in a similar manner with a few variations towards the end of the experiment. Once approximately 40 mL has collected in the beaker, turn the hot plate off and remove it from under the flask. (Be sure that the clamp supporting the flask is secure!) Do not remove the distillate collection bottle until the mixture in the flask has stopped boiling for about 5 minutes. This helps to ensure that no vapors remain in the tubing.

After the distillate collection beaker has been removed, the flask can be removed from the clamp, and the stopper/tubing apparatus can be detached. However, be sure to keep the flask covered while it is cooling so that no vapors escape. The flask mixture will need to cool quite a bit before its specific gravity can be measured. (In the meantime, finish measuring the specific gravity of the three distillate samples.) A cool water bath will help expedite the cooling process of the flask mixture. In addition, gently swirling the liquid in the flask will help release heat more quickly.

Write-up:

Chemical engineers typically perform mole balance analyses on processes as a means of checking to see that all material is accounted for. In general, this means checking to see that

Input + Generation – Output – Consumption = Accumulation

In this lab the mole balance can be thought of in terms of

Moles in original flask mixture = Moles in Distillate 1 + Moles in Distillate 2 + Moles in Distillate 3 + Moles in final flask mixture

Mole balances can be carried out for the total material in the process or for individual components. From the measured volume, specific gravity, and mass fraction of the original mixture, distillate samples, and the final mixture, you are able to calculate the moles of ethanol and water in each of these volumes of liquid. Perform a mole balance for both the ethanol and water. If necessary, propose explanations for any discrepancies in the balances.

If the assumption is made that all of the vapor produced in the flask travels through the condenser and is collected as distillate, then the mole fraction of ethanol in the distillate sample is the same as that of the vapor in the flask. (Actually, this method is not exact. The 40-mL distillate samples are collected over a period of time during which the concentration of the vapor and distillate continue to change. Thus, the mole fractions of the distillate samples reflect a sort of time-averaged concentration rather than the concentration at an instantaneous moment in time.)

The equilibrium relationship between ethanol and water determines the concentration of the vapor and liquid phases. A liquid mixture with a certain concentration of ethanol will produce a vapor phase of a unique concentration, and these concentrations are predicted by the equilibrium relationship. In this experiment, the original mixture's concentration of ethanol is known, and the first distillate sample gives an approximation of the concentration of the corresponding vapor phase. This information makes it possible to plot a point on the ethanol/water equilibrium curve. Further, since it is known how much ethanol is removed from the flask in the first distillate sample, the new concentration of ethanol in the flask can be calculated. And, as before, the second distillate sample gives an approximation of the vapor phase concentration that corresponds to the new flask mixture concentration. The same rationale can be applied for the third distillate. Therefore, with the three distillate samples, three points on the equilibrium curve can be determined. Calculate values for these three points and plot them to construct an x/y ethanol/water equilibrium curve. Compare your curve with the given reference equilibrium curve. Suggest possibilities for any differences between the two.

Distillation Lab Data Sheet

	Volume	Temperature	Specific Gravity	Mass fraction ethanol
Initial Flask Mixture				
1st distillate sample				
2nd distillate sample				
3rd distillate sample				
Final flask mixture				

Densities of ethyl alcohol (C_2H_5OH) in water

(% alcohol by weight)

%	10C	15C	20C	25C	30C	35C	40C
0			0.99823	0.99708	0.99568	0.99406	0.99225
1			636	520	379	217	034
2			453	336	194	031	.98846
3			275	157	014	.98849	663
4			103	.98984	.98839	672	485
5	098	032	.98938	817	670	501	311
6	.98946	.98877	780	656	507	335	142
7	801	729	627	500	347	172	.97975
8	660	584	478	346	189	009	808
9	524	442	331	193	031	.97846	641
10	393	304	187	043	.97875	685	475
11	267	171	047	.97897	723	527	312
12	145	041	.97910	753	573	371	150
13	026	.97914	775	611	424	216	.96989
14	.97911	790	643	472	278	063	829
15	800	669	514	334	133	.96911	670
16	692	552	387	199	.96990	760	512
17	583	433	259	062	844	607	352
18	473	313	129	.96923	697	452	189
19	363	191	.96997	782	547	294	023
20	252	068	864	639	395	134	.95856
21	139	.96944	729	495	242	.95973	687
22	024	818	592	348	087	809	516
23	.96907	689	453	199	.95929	643	343
24	787	558	312	048	769	476	168
25	665	424	168	.95895	607	306	.94991
26	539	287	020	738	442	133	810
27	406	144	.95867	576	272	.94955	625
28	268	.95996	710	410	098	774	438
29	125	844	548	241	.949922	590	248
30	.95977	686	382	067	741	403	055
31	823	524	212	.94890	557	214	.93860
32	665	357	038	709	370	021	662
33	502	186	.94860	525	180	.93825	461
34	334	011	679	337	.93986	626	257

%	10C	15C	20C	25C	30C	35C	40C
35	162	.94832	494	146	790	425	051
36	.94986	650	306	.93952	591	221	.92843
37	805	464	114	756	390	016	634
38	620	273	.93919	556	186	.92808	422
39	431	079	720	353	.92979	597	208
40	238	.93882	518	148	770	385	.91992
41	042	682	314	.92940	558	170	774
42	.93842	478	107	729	344	.91952	554
43	639	271	.92897	516	128	733	332
44	433	062	685	301	.91910	513	108
45	226	.92852	472	085	692	291	.90884
46	017	640	257	.91868	472	069	660
47	.92806	426	041	649	250	.90845	434
48	593	211	.91823	429	028	621	207
49	379	.91995	604	208	.90805	396	.89979
50		0.91776	0.91384	0.90985	0.90580	0.90168	0.89750
51	.91943	555	160	760	353	.89940	519
52	723	333	.90936	534	125	710	288
53	502	110	711	307	.89896	479	056
54	279	.90885	485	079	667	248	.88823
55	055	659	258	.89850	437	016	589
56	.90831	433	031	621	206	.88784	356
57	607	207	.89803	392	.88975	552	122
58	381	.89980	574	162	744	319	.87888
59	154	752	344	.88931	512	085	653
60	.89927	523	113	699	278	.87851	417
61	698	293	.88882	446	044	615	180
62	468	062	650	233	.87809	379	.86943
63	237	.88830	417	.87998	574	142	705
64	006	597	183	763	337	.86905	466
65	.88774	364	.87948	527	100	667	227
66	541	130	713	291	.86863	429	.85987
67	308	.87895	477	054	625	190	747
68	074	660	241	.86817	387	.85950	407
69	.87839	424	004	579	148	710	266
70	602	187	.86766	340	.85908	470	025
71	365	.86949	527	100	667	228	.84783
72	127	710	287	.85859	426	.84986	540
73	.6888	470	047	618	184	743	297
74	648	229	.85806	376	.84941	500	053
75	408	.85988	564	134	698	257	.83809
76	168	747	322	.84891	455	013	564
77	.85927	505	079	647	221	.83768	319
78	685	262	.84835	403	.83966	523	074

79	442	018	590	158	720	277	.82827
%	**10C**	**15C**	**20C**	**25C**	**30C**	**35C**	**40C**
80	197	.84772	344	.83911	473	029	578
81	.4950	525	096	664	224	.82780	329
82	702	277	.83848	415	.82974	530	079
83	453	028	599	164	724	279	.81828
84	203	.83777	348	.82913	473	027	576
85	.83951	525	095	660	220	.81774	322
86	697	271	.82840	405	.81965	519	067
87	441	014	583	148	708	262	.80811
88	181	.82754	323	.81888	448	003	552
89	.82919	492	062	626	186	.80742	291
90	654	227	.81797	362	.80922	478	028
91	386	.81959	529	094	655	211	.79761
92	114	688	257	.80823	384	.79941	491
93	.81839	413	.80983	549	111	669	220
94	561	134	705	272	.79835	393	.78947
95	278	.80852	424	.79991	555	114	670
96	.80991	566	138	706	271	.78831	388
97	698	274	.79846	415	.78981	542	100
98	399	.79975	547	117	684	247	.77806
99	094	670	243	.78814	382	.77946	507
100	.79784	360	.78934	506	075	641	203